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DIGITAL SIMULATION OF DIFFERENTIAL PULSE POLAROGRAPHY WITH INCR--ETC(U)
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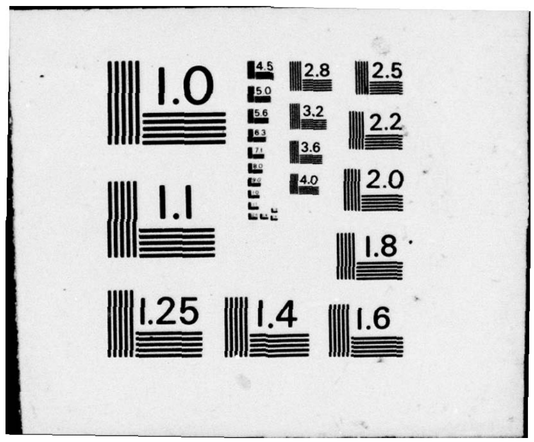
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TECHNICAL REPORT NO. 1

DIGITAL SIMULATION OF DIFFERENTIAL PULSE POLAROGRAPHY WITH INCREMENTAL TIME CHANGE

by

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Colorado State University
Department of Chemistry
Fort Collins, Colorado 80523

November, 1976

404 992

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER Technical Report No. 1	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER 14 TR-1
4. TITLE (and Subtitle) 6 Digital Simulation of Differential Pulse Polarography with Incremental Time Change.		5. TYPE OF REPORT & PERIOD COVERED 9 Interim rept., 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) 10 James W./Dillard, John A./Turner ~~and~~ R. A./Osteryoung		8. CONTRACT OR GRANT NUMBER(s) N00014-77-C-0004 15 N00014-77-C-0004
9. PERFORMING ORGANIZATION NAME AND ADDRESS Department of Chemistry Colorado State University Fort Collins, CO 80523		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS Chemistry Program Office of Naval Research Arlington, Virginia 22217		12. REPORT DATE 11 November, 1976 13. NUMBER OF PAGES 28 12 30p.
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) Office of Naval Research Resident Representative Suite 210, 6740 E. Hampden Avenue Denver, CO 80222		15. SECURITY CLASS. (of this report) Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for Public Release; Distribution Unlimited

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

prepared for publication in Analytical Chemistry

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Pulse polarography; digital simulation

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Explicit finite difference simulations of TAST, normal pulse and differential pulse polarography for the reversible case have been developed for conserving computer time. The method involves changing the time increment during the simulation. The results are shown to conform to accepted theory and include sphericity and the DME compression of the diffusion layer due to the drop expansion in area. The DC faradaic distortion of differential pulse ~~pulse~~ is also studied. The simulation gives a factor of five savings in computer time over simulators of the standard type.

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BRIEF

Finite difference simulations of TAST, normal and differential pulse polarography are presented which reduce computer execution time fivefold. Sphericity, D. C. distortion and DME compression are included. Simulated results are shown to be within 0.5% of theoretical.

ABSTRACT

Explicit finite difference simulations of TAST, normal pulse and differential pulse polarography for the reversible case have been developed for conserving computer time. The method involves changing the time increment during the simulation. The results are shown to conform to accepted theory and include sphericity and the DME compression of the diffusion layer due to the drop expansion in area. The DC faradaic distortion of differential pulse is also studied. The simulation gives a factor of five savings in computer time over simulators of the standard type.

Explicit finite difference simulations of electrochemical processes have been described by Feldberg (1). In this method, numerical arrays are used to represent the concentration profiles of the electroactive species as a function of distance from the electrode surface. Time and space are divided into discrete time and space increments. Operations representative of Fick's laws of diffusion, charge transfer and homogeneous chemical reactions manipulate the concentration arrays within the space-time grid. Because this method is explicit, stable solutions will be obtained only if the following condition is maintained:

$$\frac{\Delta t \cdot D}{\Delta x^2} < 0.5$$

where Δt is the time increment, Δx is the space element and D is the diffusion coefficient. In Feldberg's model, the Δx and Δt increments are uniformly spaced and represented in the computer program by a single cycle in a "do loop." Unfortunately, maximum resolution requires large numbers of space and time elements when simulating long times or fast reactions. Joslin et al (2) have described a method of mapping the space grid using a non-linear function. Near the electrode surface where the concentration profile is critical, the space elements are closely spaced and increase in size with distance from the electrode. Booman et al (3) and Winograd (4) have used the implicit form of finite difference to conserve computer time.

With normal pulse and differential pulse polarography, the most important part of drop life is the last few msec when the voltage pulse is applied. In previous finite difference simulations of differential pulse polarography (5), time was incremented in accordance with the resolution required for the perturbation at the end of drop life (pulse application). Many unnecessary iterations during drop delay time were required and the resulting lengthy programs prohibited use of this method for many studies. The object of this investigation was to develop a finite difference simulator that could use large time increments during drop growth where resolution is not critical and change the increment just prior to pulse application so that the needed time resolution could be obtained for the pulse measurement. Such finite difference simulations have been written for TAST, normal pulse and differential pulse polarography. These programs give a factor of five savings in computer execution time for an 1800 loop simulation over previous simulators of the standard Feldberg type. The results for the reversible case are in excellcnt agreement with accepted theory and will be presented here.

METHOD

The finite difference simulators discussed here are patterned after Feldberg and Ruzic (6) but incorporate important changes. Simulations should emulate the true experimental output as closely as possible. For this reason, the simulated instantaneous flux is normalized to reflect the $t^{1/6}$ dependence of current as a function of time during drop life at a DME. This dependence is necessary to incorporate the DC concentration-independent distortion in current of the differential pulse polarographic peak. This normalization also reveals the $\sqrt{7/3}$ compression factor in the polarographic diffusion limited current. The most important modification to the simulations is the capability to change the Δt increment at will. A given drop time can be divided into time increments or execution do loops. Obviously, the more loops utilized, the better the resolution of time over drop life. The simulations used here are initiated using a maximum number of time units (MNTU) equal to 50 over the life of one drop. A flow chart indicating the order of program execution is illustrated in Figure 1. At an appropriate time loop (loop 46) prior to pulse application, the simulator is converted to a MNTU equal to 450. The simulator then continues from loop 415 through 450. Therefore, a 450 loop experiment can be simulated in just 82 loops.

All parameters which are a function of MNTU must be converted to their appropriate value in the 450 loop experiment. Table I indicates the variables which must be changed. The most important and sensitive transformation involves the reactant and product concentration profiles. During program conversion to the new Δt basis, the concentration profiles are fit to a ninth degree polynomial and the coefficients are used in calculation of the new expanded concentration profile. In order to refine this operation, the resulting concentration values are corrected by a small volume element weighted factor. Because the volume elements considered are a function of the $\sqrt{MNTU}$, the choice of 50 and 450 was made so that the square root of their ratio would be an odd integer. Other MNTU values are acceptable as long as the square root and odd integer relationship is met. This insures symmetrical conversion from one set of volume elements to the other set. When a comparison is made between the concentration profile just after the Δt time change in a simulation which emulates a 450 loop experiment and the concentration profile at the same relative time in a simulation of 450 actual time loops, the two profiles are indistinguishable. Thus, the technique reaches the same concentration profile in less time than required for the standard simulation.

EXPERIMENTAL

The minicomputer system used for these simulations consists of a Digital Equipment Corporation PDP-12 minicomputer with 24K of core (12 bit words), scope, x-y plotter, dual magnetic tape, 1.6 megaword disk and hardware floating point processor (FPP 12). The FPP 12 operates in a parallel processing mode, performing the arithmetic operations leaving the CPU free to display the concentration profiles and execute calculations simultaneously. This feature is not only a very useful instructive feature, but is also a powerful "debugging" tool. Although a hardware floating point processor is not a requirement for the running of these programs, it does greatly speed up the execution of FORTRAN IV.

In single precision mode, the system carried six significant digits, double precision gives 17. Except for the polynomial fitting routine, all programs reported here used single precision. The program consists of a core resident mainline and a group of stored subroutines. These subroutines reside on a magnetic disk and are overlayed into core when called. This overlay structure allows programs to be run that ordinarily would be too large to fit into available core.

The results presented here are for the expanding planar model. The current-time profile for a single drop using the modified simulator is illustrated in Figure 2. An enlargement of the Δt time change region is also shown. The current-time profile is continuous over drop life with only slight oscillation occurring at the Δt time change. Figure 3 shows a plot of log current versus log time for the above-mentioned current-time profile and an accompanying enlargement. The expected slope of 0.167 is followed over all but the first few points.

TAST POLAROGRAPHY

The TAST measurement was taken on the (T-1) time loop prior to drop termination and the scanning wave form was a staircase of 10 mV step height. The resulting current voltage curve for n = 1 is in Figure 4a. The current voltage curves for n = 2 and 3 also behaved as expected. In Figure 4b an E versus log $(I/I_d - I)$ plot for the n = 1 case verifies the electron transfer reversibility. Table II summarizes the TAST results and compares the simulator diffusion currents with the expected value of $n \cdot \sqrt{7/3}$.

NORMAL PULSE POLAROGRAPHY

The pulse time selected for this study was 50 msec, with current measurement occurring on the (T-1) time loop before the end of drop life. Figure 5 shows the current time profile for one drop as the potential is pulsed from before the wave to the diffusion plateau. The log-log plot in Figure 5b of current versus time during pulse application gave the expected -0.5 slope, as did all cases irrespective of pulse potential on the wave. Normal pulse data compared favorably with the expected values calculated from the TAST results (Table III).

DIFFERENTIAL PULSE POLAROGRAPHY

The pulse time was 50 msec and the difference measurement was made between the current just prior to pulse application and the current at the end of drop life. Figure 6 illustrates the pulse decay current at a potential -50 mV from E° for a pulse height of 50 mV. In Figure 7 the log-log plot of the pulse decay current with time is examined as a function of scan potential at an E_{pulse} of 50 mV. The slope depends on the degree of concentration polarization prior to pulse application. This slope changes from -0.5 for potentials before $E_{1/2}$ to a slope of 1.9×10^{-3} on the diffusion plateau. The nearly horizontal slope results because the time is referenced with respect to the start of pulse application. If time is plotted with respect to initiation of the drop, then the log-log plot has the familiar slope of 0.167 as is shown in Figure 8. The DC faradaic effect described by Christie et al (7) is clearly demonstrated in Figure 9. This distortion is due to the increase in the DC current component because of drop growth over the short pulse time. The differential pulse data is compared to values derived from Parry and Osteryoung (8) and calculated from the normal pulse data in Table IV. The results are within the uncertainty in the simulation.

SPHERICITY

Sphericity (S) is a dimensionless parameter defined by the following expression:

$$S = \frac{\sqrt{Dt}}{r}$$

where t is the drop time and r is the drop radius.

From experimentally determined quantities, S can be calculated and the dimensionless value used in the simulations to incorporate the appropriate sphericity correction. A complete description and derivation of the theory is described by Newman (9). The effects of sphericity for selected values of S are summarized in Table V through VII for TAST, normal pulse and differential pulse polarography. The limiting current values for normal pulse are lower than calculated from the TAST data with sphericity because of the short time measurement. As is expected for short time pulse measurements, sphericity has little effect on normal pulse and differential pulse polarograms.

DISCUSSION

The experimental time to simulator loop relationship determines the resolution of the simulation. For a one-second drop time experiment simulated by a modified 450 loop simulation, the resolution during the delay time was 20 msec/loop and during pulse application was 2.2 msec/loop. This can be translated into an uncertainty in normalized current of 0.16 units and an uncertainty in potential of 0.4 mV. Better resolution can be achieved by increasing the MNTU value at the expense of execution time.

The program used in this work executes a 450 loop simulation in only 82 time loops per drop at approximately 35 seconds computation time per drop. The comparable program not including the time saving features executed the 450 loop simulation in just over two minutes per drop. This is a dramatic savings in time for the execution of a 100 drop polarogram.

Application of this unique method of programming is being used in this laboratory in simulations involving in situ generation at electrode surfaces and electron transfer reactions involving homogeneous kinetics. Also, quasi-reversible and irreversible heterogeneous kinetics are being studied using this method. With faster and larger minicomputers becoming available, computer simulation of reactions will become an increasingly important tool in the experimental and theoretical laboratory.

CREDIT

This work was supported in part by the National Science Foundation under grant number CHE-7610445 and by the Office of Naval Research under contract number N00014-77-C-004.

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TABLE I. PROGRAM VARIABLE MODIFICATIONS

ΔV	=>	$4\pi\ D^{3/2}\ t_f^{\ 1/2}/(3\ S_f^{\ 3})$
V	=>	$t\ \Delta V$
r'	=>	$(3V/(4\pi))^{1/3}$
r	=>	$(3(t-1)\ \Delta V/(4\pi))^{1/3}$
S_t	=>	$(Dt)^{1/2}/r$
i_{max}	=>	$5\ (Dt_f)^{1/2}$
i_{DME}	=>	$4\ (Dt)^{1/2} + 3$
J_N	=>	$0.5\ (J_A + J_A')\ (\pi\ t_f/D)^{1/2}$
K_{hs}	=>	$RATE\ (D * DROPTM/(t_f * DFCOEF))^{1/2}$

t = new time

t_f = new final time

RATE = experimental rate

DROPTM = experimental drop time

DFCOEF = experimental diffusion coefficient

TABLE II. TAST POLAROGRAPHY

	E versus log $(I/I_d - I)$		I_d	
	Slope	y-intercept	Simulator	Theory
n=1	-0.0590	1.5×10^{-4}	1.53	1.53
n=2	-0.0296	1.2×10^{-6}	3.06	3.05
n=3	-0.0197	-1.1×10^{-6}	4.60	4.58

TABLE III. NORMAL PULSE POLAROGRAPHY

	Simulator		Theory
	$E_{1/2}$	I_d	I_d
n=1	0.0	4.51	4.48
n=2	0.0	9.02	8.96
n=3	0.0	13.54	13.47

TABLE IV. DIFFERENTIAL PULSE POLAROGRAPHY

	Pulse Height mV	E_{pk} mV vs E°	I_{pk}		Half Width, mV	
			Simulator	Theory	Simulator	Theory
n=1	50	25.0	2.04	2.04	99.3	99.1
	100	50.0	3.38	3.38	123.6	123.4
	150	75.0	4.05	4.05	160.1	160.1
n=2	50	25.0	6.77	6.77	61.8	61.7
	100	50.0	8.66	8.66	102.1	102.0
n=3	50	25.0	12.15	12.17	53.1	53.4
	100	50.0	13.46	13.47	100.2	100.2

TABLE V. TAST POLAROGRAPHY WITH SPHERICITY

	S	E versus log $(I/I_d - I)$		I_d	
		Slope	y-intercept	Simulator	Theory
n=1	0.1	-0.0591	4.7×10^{-5}	1.69	1.69
	0.2	-0.0590	1.6×10^{-4}	1.85	1.84
	0.3	-0.0591	4.8×10^{-5}	2.02	2.00
n=2	0.1	-0.0296	1.7×10^{-7}	3.38	3.36
	0.2	-0.0296	5.0×10^{-7}	3.71	3.68
	0.3	-0.0296	-3.1×10^{-7}	4.05	3.99
n=3	0.1	-0.0197	-6.9×10^{-7}	5.07	5.05
	0.2	-0.0197	6.8×10^{-7}	5.57	5.52
	0.3	-0.0197	6.8×10^{-7}	6.08	6.00

TABLE VI. NORMAL PULSE POLAROGRAPHY WITH SPHERICITY

	S	$E_{1/2}$	I_d		
			Simulator	Theory I[a]	Theory II[b]
n=1	0.1	0.0	4.69	4.48	4.95
	0.2	0.0	4.86	4.48	5.42
	0.3	0.0	5.04	4.48	5.91
n=2	0.1	0.0	9.37	8.96	9.89
	0.2	0.0	9.73	8.96	10.86
	0.3	0.0	10.08	8.96	11.86
n=3	0.1	0.0	14.06	13.47	14.84
	0.2	0.0	14.59	13.47	16.31
	0.3	0.0	15.13	13.47	17.80

[a]Calculated without sphericity

[b]Calculated with sphericity

TABLE VII. DIFFERENTIAL PULSE POLAROGRAPHY WITH SPHERICITY

	S	Pulse Height mV	E_{pk} mV vs E°	I_{pk}		Half Width, mV	
				Simulator	Theory	Simulator	Theory
n=1	0.1	50.0	25.0	2.12	2.12	99.2	99.1
	0.2	50.0	25.0	2.20	2.20	99.2	99.1
	0.3	50.0	25.0	2.28	2.28	99.2	99.1
n=2	0.1	50.0	25.0	7.03	7.03	61.8	61.7
	0.2	50.0	25.0	7.30	7.30	61.8	61.7
	0.3	50.0	25.0	7.56	7.56	61.8	61.7
n=3	0.1	50.0	25.0	12.62	12.64	53.4	53.4
	0.2	50.0	25.0	13.10	13.12	53.4	53.4
	0.3	50.0	25.0	13.58	13.60	53.4	53.4

FIGURE CAPTIONS

Figure 1 Finite difference simulation flow chart.

Figure 2 Single drop current-time profile of TAST polarography. Drop time = 1.0 sec; E = -300 mV. (A) Total drop. (B) Expansion of 0.8 to 1.0 sec region.

Figure 3 Single drop log current-log time plot of TAST polarography. Drop time = 1.0 sec; E = -300 mV. (A) Total drop; Slope = 0.167 (-). (B) Expansion of 0.8 to 1.0 sec region.

Figure 4 TAST polarography. (A) Current-potential profile. Drop time = 1.0 sec; Step height = 5 mV. (B) E vs log (I/Id-I).

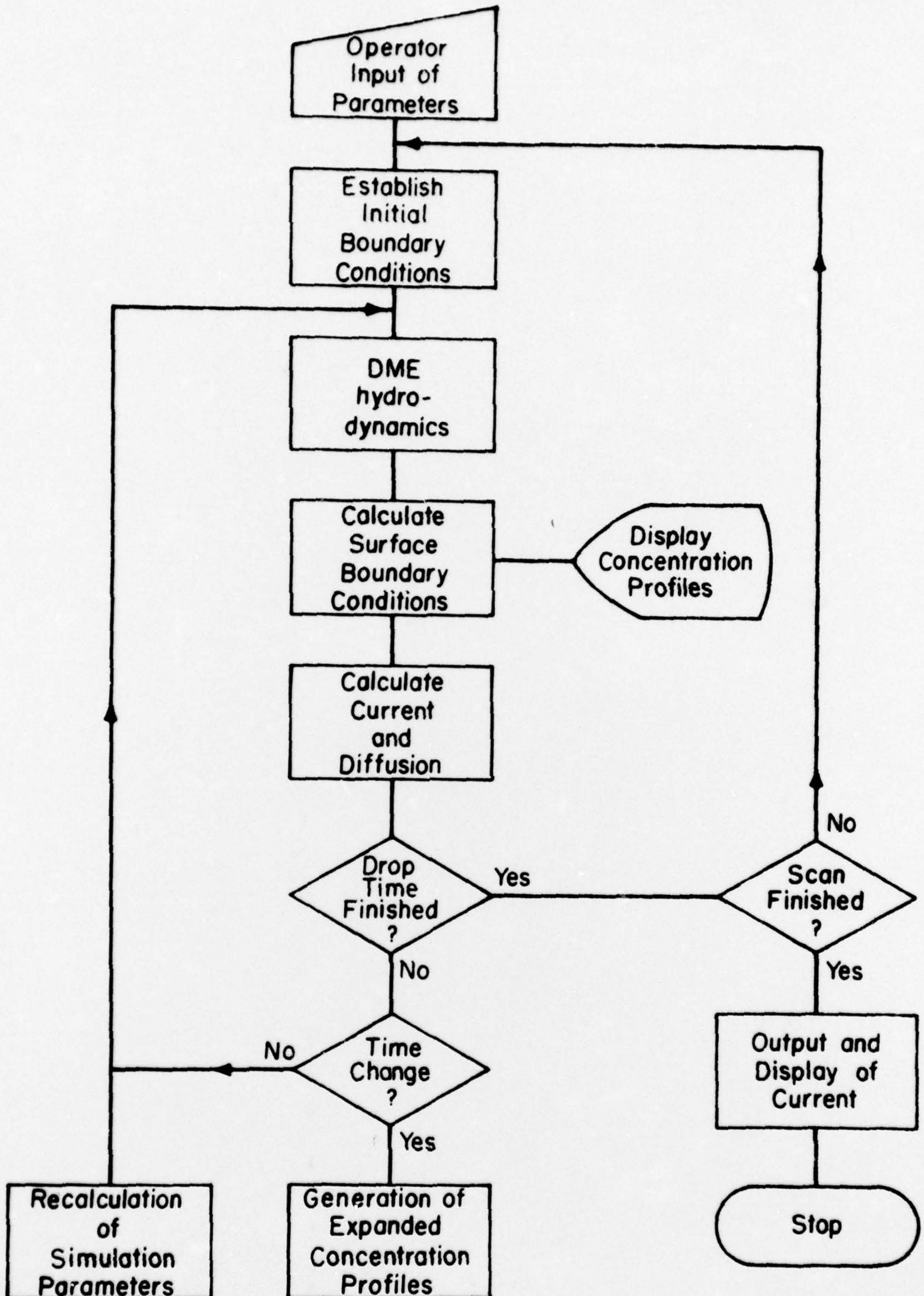
Figure 5 Single drop current-time profile of normal pulse polarography. Drop time = 1.0 sec; Pulse time = 50 msec; E = 300.0 mV; E_{pulse} = 600.0 mV. (A) Current-time profile expansion of 0.8 to 1.0 sec region. (B) Log current-log time plot of pulse decay. Slope = 0.5 (-).

Figure 6 Single drop current-time profile of differential pulse polarography. Drop time = 1.0 sec; Pulse time = 50 msec; E = -50 mV; E_{pulse} = 50 mV. (A) Total drop. (B) Expansion of 0.8 to 1.0 sec region.

Figure 7 Pulse decay log current-log time plot of differential pulse polarography. Drop time = 1.0 sec; Pulse time = 50 msec; E_{pulse} = 50 mV. (A) E = 100.0 mV; Slope = 0.5 (-). (B) E = 0.0 mV; Slope = 0.5 (-). (C) E = -100 mV; Slope = 1.9×10^{-3}. (D) E = -300 mV; Slope = 1.9×10^{-3}.

Figure 8 Single drop current-time profile of differential pulse polarography. Drop time = 1.0 sec; Pulse time = 50 msec; E_{pulse} = 50 mV; E = -300 mV. (A) Total drop. (B) Total log current-log time; Slope = 0.5 (-).

Figure 9 Differential pulse polarography with sphericity. Drop time = 0.5 sec; Pulse time = 40 msec; Step height = 10 mV; E_{pulse} = 10 mV. (A) S = 0. (B) S = 0.3.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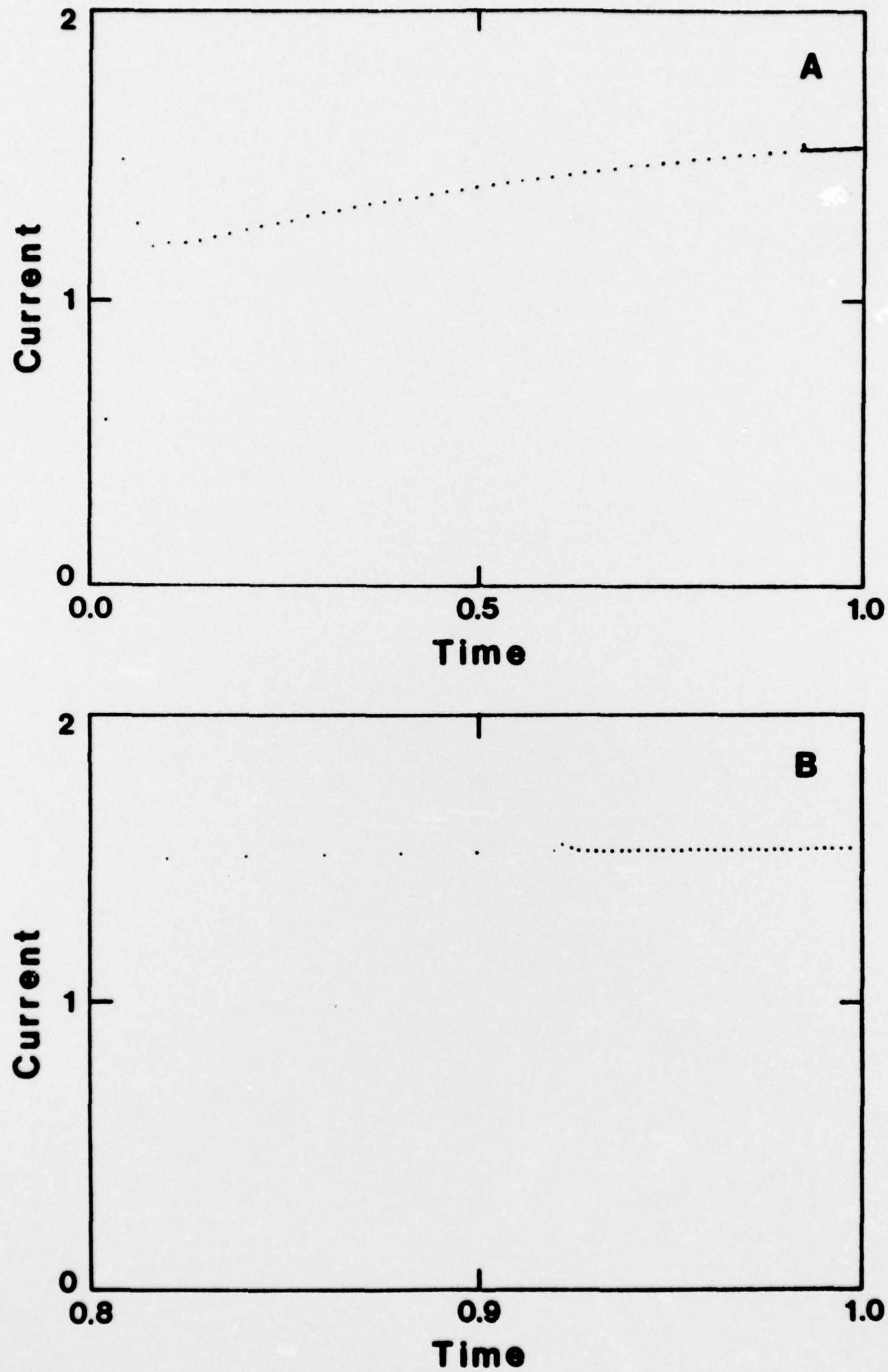

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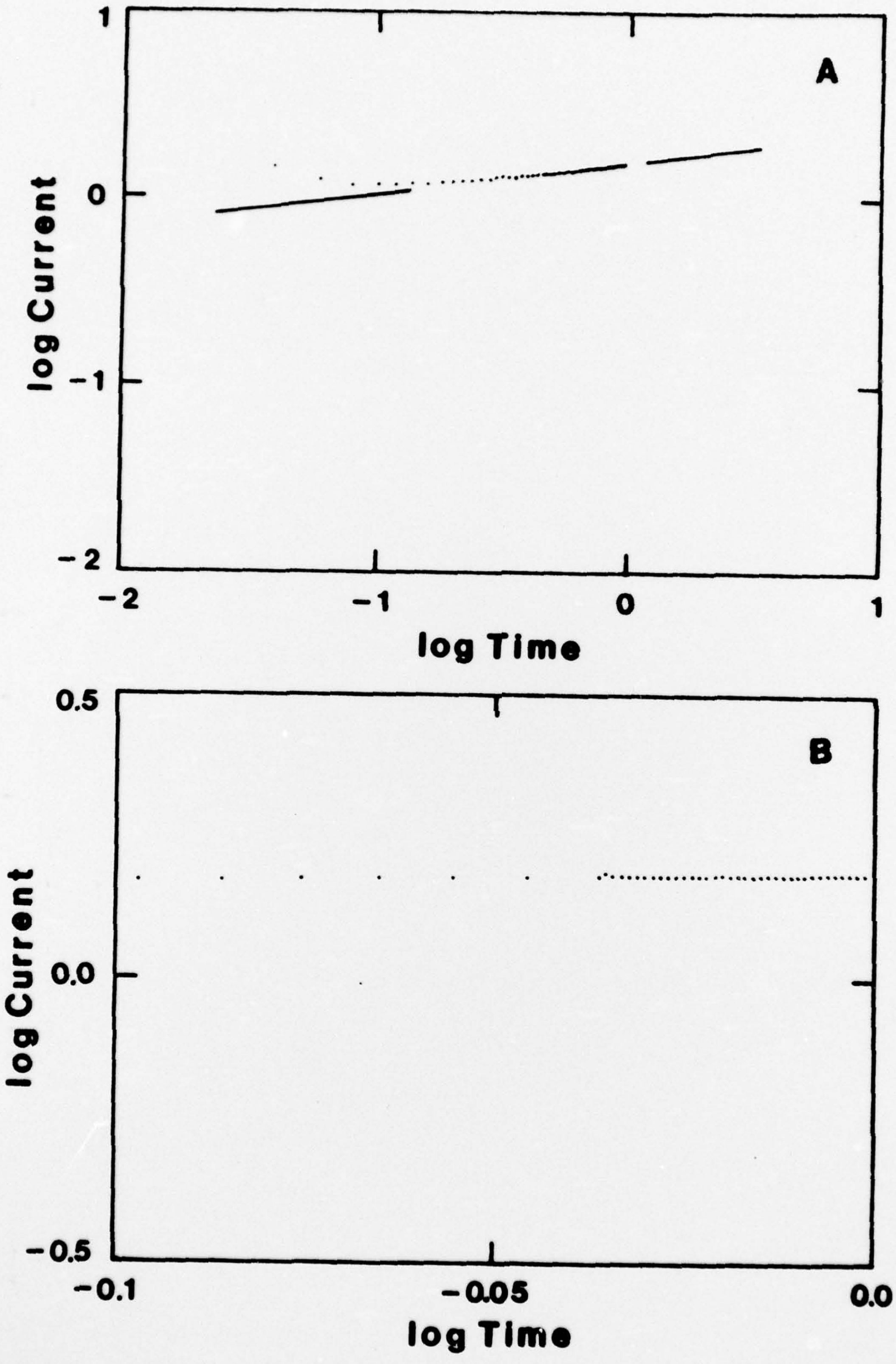

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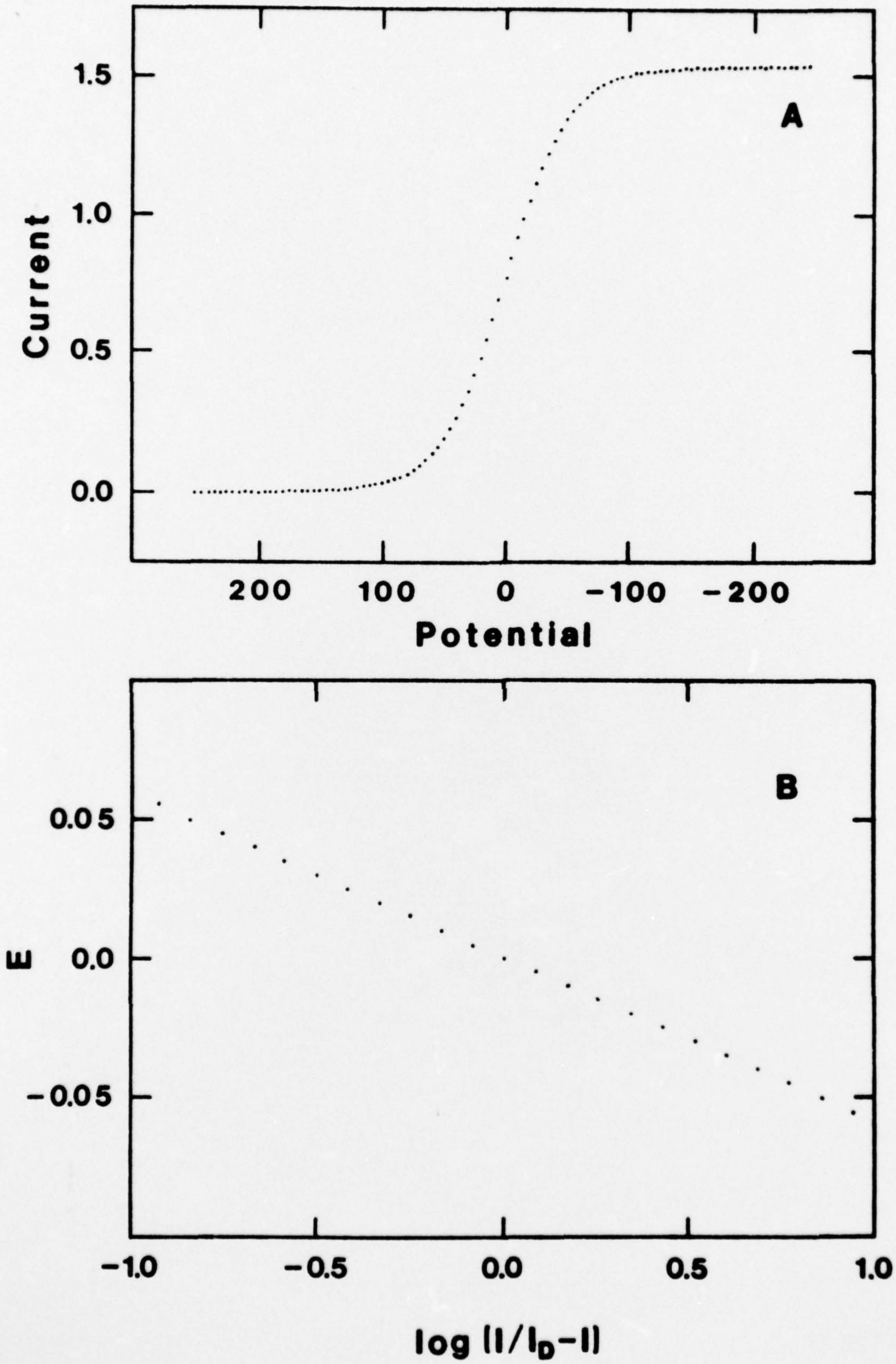
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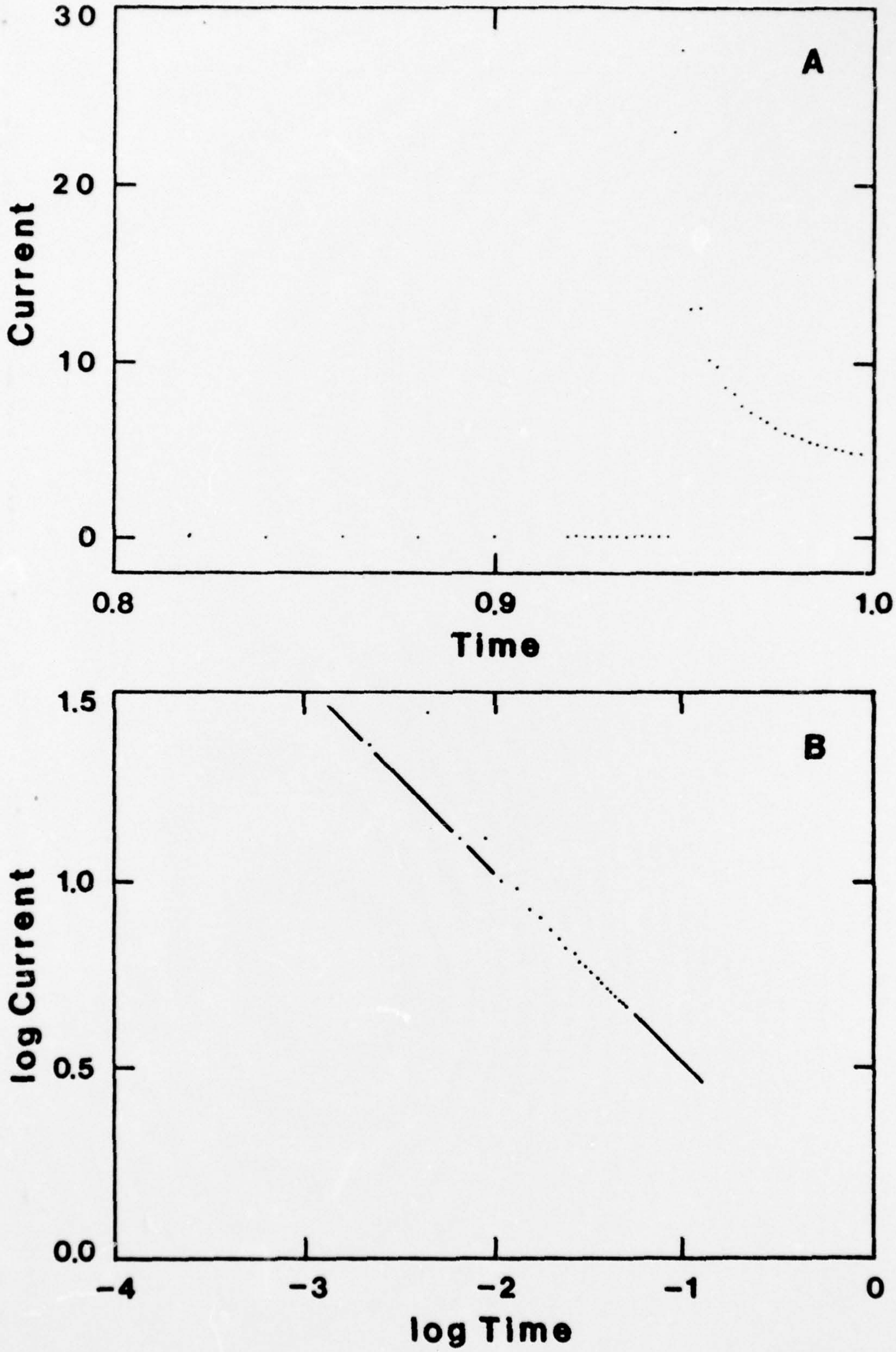
FIGURE 4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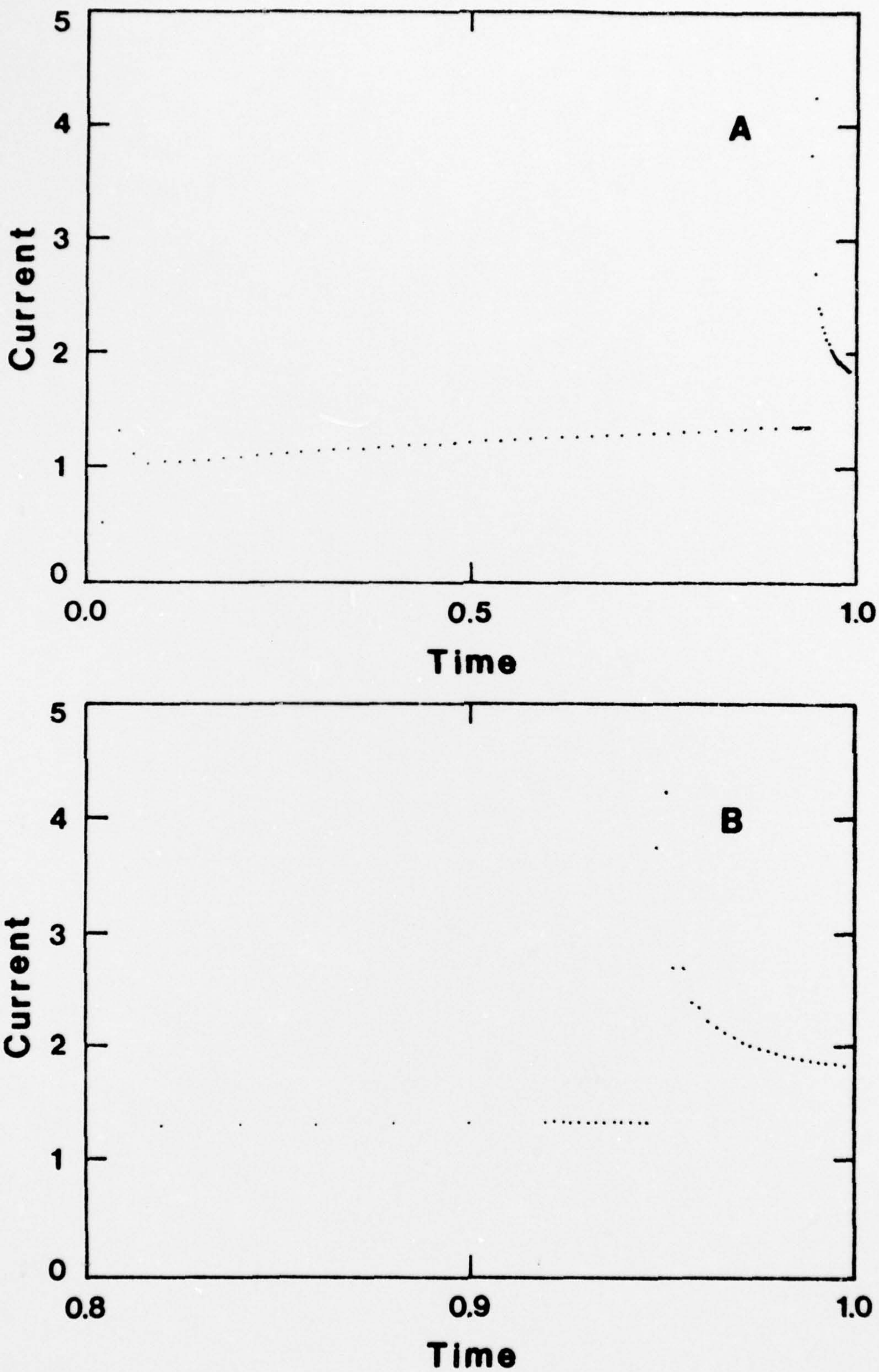
0
log Time

FIGURE 5



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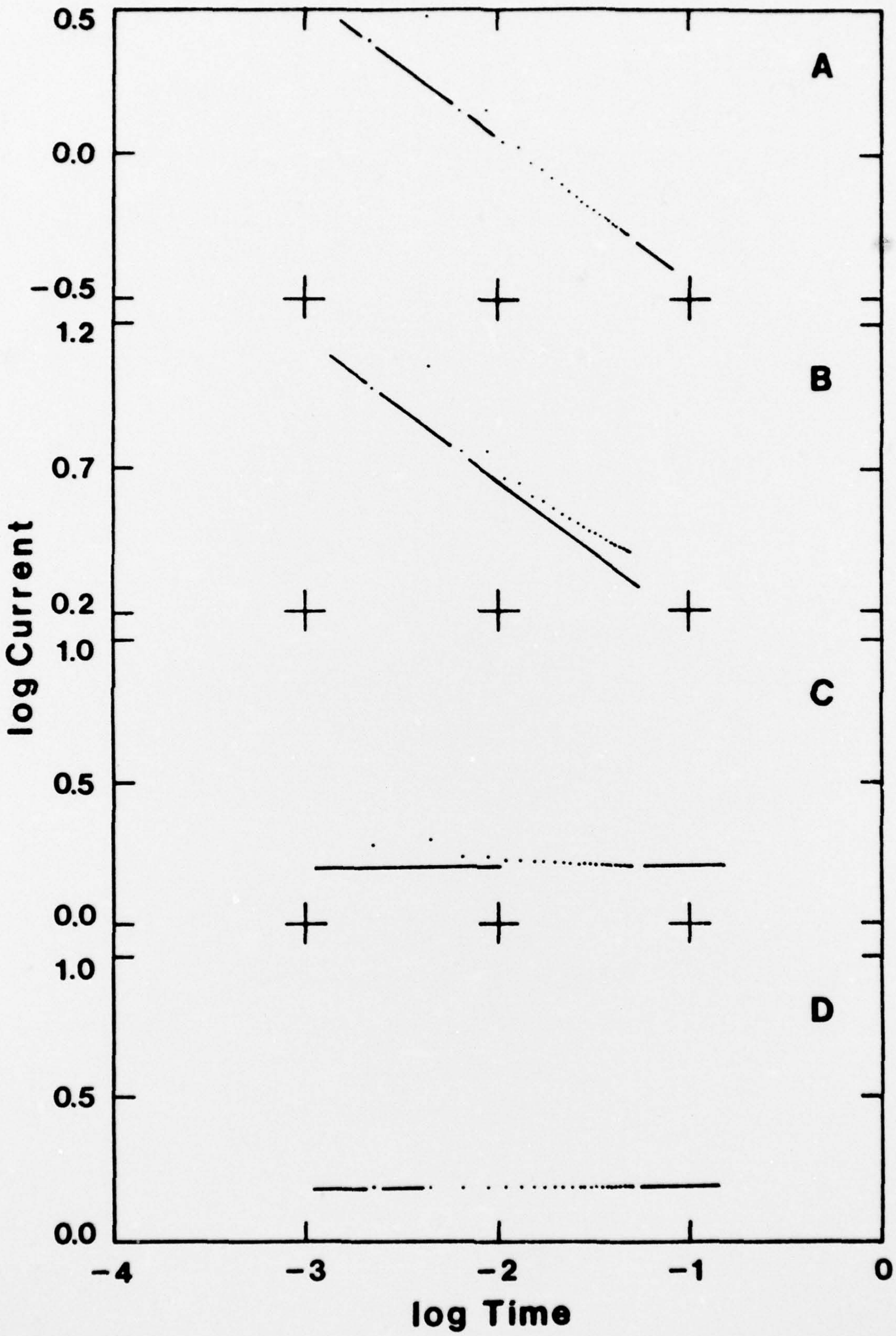
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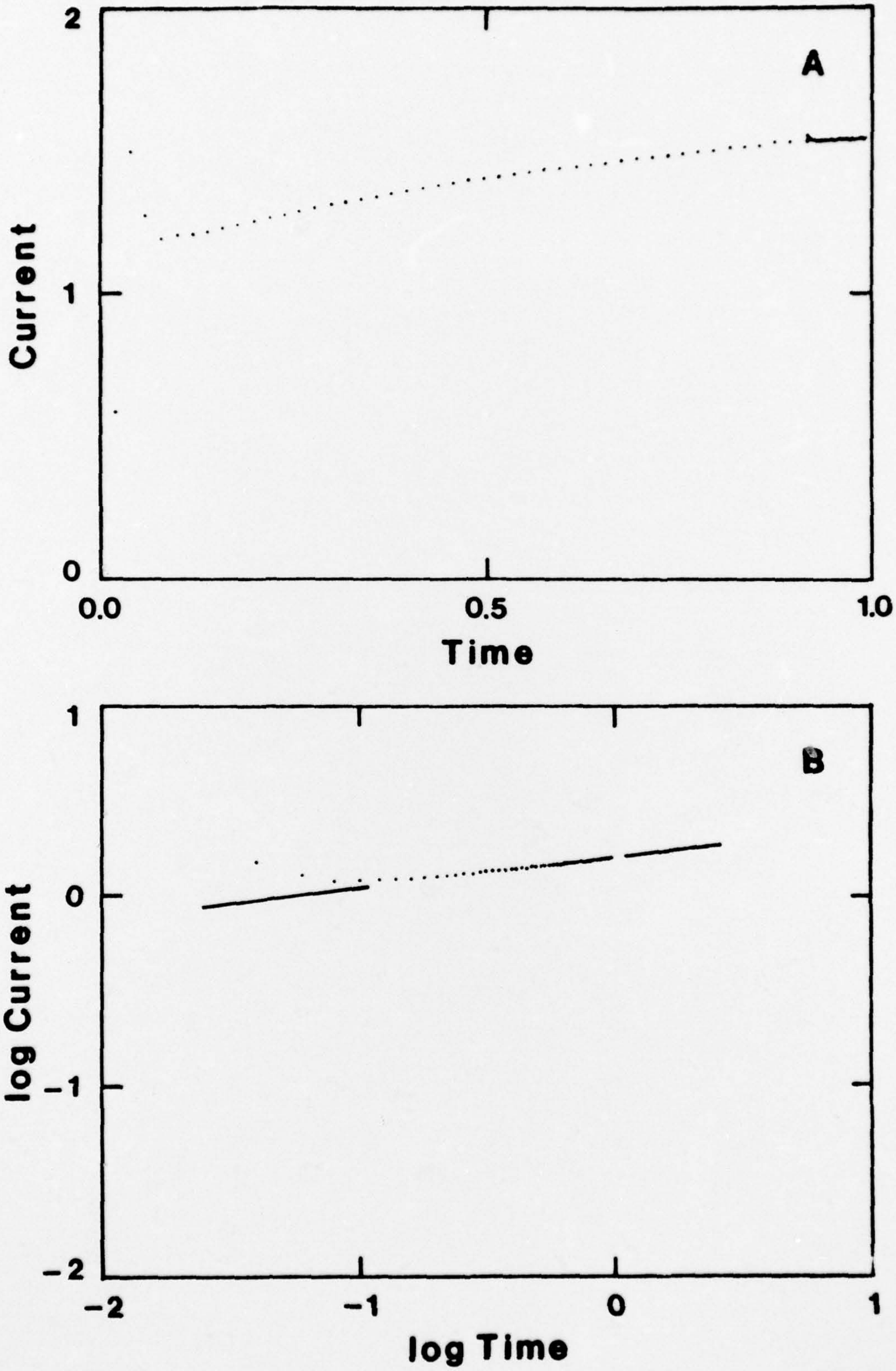
FIGURE 7



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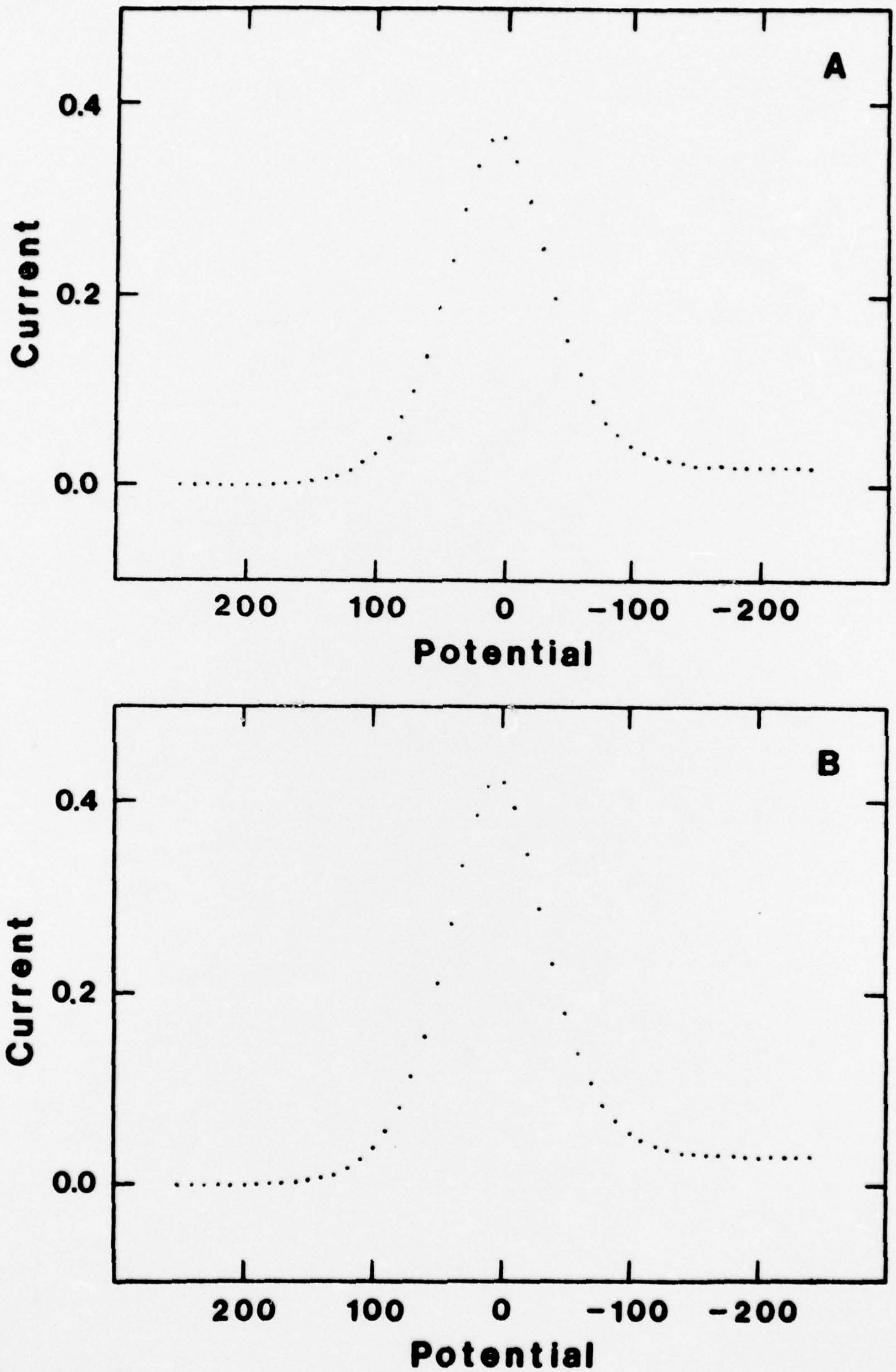
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FIGURE 9

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